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FABRICATION OF CERMETS OF URANIUM NITRIDE AND TUNGSTEN OR MOLYBDENUM FROM MIXED POWDERS AND FROM COATED PARTICLES

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ABSTRACT

Cermets containing uranium nitride (UN) particles dispersed in a matrix of tungsten (W) or molybdenum (Mo) were fabricated by cold-pressing and sintering mixed powders and by hot isostatic compaction of metal-coated UN particles. The effects of powder particle size and sintering conditions (time, temperature, and atmosphere) on the microstructure and density of cermets containing 50 volume percent of UN were studied. Some sintered cermets were further densified by hot pneumatic impaction. Spherical UN particles coated with W or Mo were consolidated by hot isostatic compaction to produce dense cermets containing from 50 to 70 volume percent of UN.

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SUMMARY

Fuel elements consisting of uranium nitride (UN) particles dispersed in either a tungsten (W) or molybdenum (Mo) matrix are attractive for use in future space nuclear power reactors. In this study, two methods of fabricating such fuel elements were investigated.

The sintering characteristics of pressed powder mixtures of UN (50 vol. %) and W or Mo were investigated. By varying the powder particle size and the sintering conditions (time, temperature, and atmosphere), cermets with densities of 80 to 98 percent of theoretical were produced. In general, the pores in these cermets were interconnected to the specimen surfaces when the density did not exceed about 90 percent of theoretical.

Some of the sintered cermets were further densified by hot pneumatic impaction. A sintered W-UN cermet was densified from 88 to over 98 percent density by hot pneumatic impaction.

Cermets also were fabricated from relatively large (75 to 150 μm in diameter) metal-coated spherical UN particles. The feasibility of preparing spherical UN particles by a plasma torch process was demonstrated by two vendors. But problems were encountered in consistently controlling density, oxygen contamination, and stoichiometry of the UN particles. A thin coating of W or Mo was deposited on the UN particles by an electron beam vaporization process. Although microcracks were present in these coatings, these cracks disappeared after the particles were hot isostatically consolidated into cermets. Cermets exhibiting 50 to 70 volume percent of UN, continuous metal matrices around the UN particles, and densities exceeding 97 percent of theoretical were successfully produced.

No evidence of chemical incompatibility between UN and the metals was observed provided that nitrogen was present over the cermets at temperatures above 1500° C. When nitrogen was not present, evidence of UN decomposition and liquid-phase sintering was found. This decomposition and liquid-phase sintering appeared to be more severe in Mo-UN than in W-UN cermets.

INTRODUCTION

Uranium nitride (UN) is an attractive fuel for use in space power reactors. The advantages of UN (refs. 1 to 3) over other fuels (e.g., uranium dioxide (UO_2)) include its relatively high uranium density, high thermal conductivity, low thermal expansivity, and good chemical compatibility with reactor coolant metals such as lithium. In addition, UN exhibits a relatively high melting point and good dimensional stability during irradiation.

The major disadvantage of UN is its tendency to decompose into uranium (U) and nitrogen (N_2) at elevated temperatures in the absence of a N_2 overpressure. For example, the decomposition pressure of N_2 over UN at 1400° C is 10^{-8} atmosphere (ref. 4). The presence of free U is particularly undesirable because of grain boundary attack on cladding metals such as molybdenum (Mo) and tungsten (W) (refs. 5 and 6). However, the amount of U formed may not be significant provided that the reactor operating temperature is not excessive and a small N_2 pressure is present.

Although the thermal conductivity of UN is higher than that of other potential ceramic fuels, unwanted fuel temperature gradients may still develop. Therefore, it may be necessary to disperse UN particles in a continuous metal matrix to reduce thermal gradients. Such a matrix also could provide increased fuel strength and better fission gas retention capabilities. Tungsten and Mo are attractive matrix metals in UN cermets because of their relatively good high temperature strength, thermal conductivity, and compatibility with UN (refs. 7 to 9).

The amount of porosity desired in cermet fuel elements is dependent on the amount of fuel burnup required in a reactor. Reactors requiring sufficiently low fuel burnups (<2 at. % U) can rely on containment of fission gases by relatively high-strength, dense, cermet fuels; on the other hand, reactors requiring higher burnups may require porous cermet fuels to permit fission gas venting and to accommodate fuel swelling.

The purpose of this study was to fabricate and characterize W-UN and Mo-UN cermets. Two fabrication processes were used to produce cermets with various densities (from 80 to 100 percent of theoretical) and fuel loadings (50 to 70 vol. % of UN) which may be required in reactor applications. These processes were (1) cold-pressing and sintering of powder mixtures and (2) hot isostatic consolidation of metal-coated UN particles.

The metallographic, density, and chemical analysis results of the fabrication and characterization of UN cermets are presented in this report. The first section presents the results on the sintered powder cermets, and the following section presents the results on the coated-particle cermets.

SINTERED POWDER CERMETS

Fabrication of Sintered Cermets

Cermets containing 50 volume percent of UN were fabricated by cold-pressing blended powders into rods and then sintering these rod specimens. Sintering characteristics were studied by varying the powder particle sizes and the sintering time, temperature, and atmosphere (mixtures of argon (Ar) and N_2). The size and distribution of pores and the chemical compatibility of the sintered cermets were also studied.

Preparation of cold-pressed powder mixtures. - Powder mixtures were prepared from two lots of depleted UN powder obtained from the Oak Ridge National Laboratory and from W and Mo powders obtained from commercial vendors. The metal powders were heated in dry hydrogen (H_2) at 800° C for 18 hours to reduce the oxygen (O_2) contents. Both the metal and UN powders were stored and handled in Ar at all times until sintering was completed. The purities, surface area, and UN stoichiometry of these powders are given in table I. Although the two UN lots exhibited no significant difference in stoichiometry (see the appendix for the precision of chemical analysis), the second lot exhibited a finer particle size and a higher O_2 content.

Five types of powder mixtures containing nominally 50 volume percent of UN and 50 volume percent of either W or Mo were prepared. No binder material was added to the powder mixtures. Each mixture was blended for 4 hours in a V-blender and then vibratorily compacted into a rubber tube. The tubes were sealed and cold isostatically pressed at 70 000 psi (500 MN/m^2) to form 1/4-inch- (0.6-cm-) diameter rods. The resultant density of these rod specimens was about 70 percent of theoretical. The fuel (UN) loadings of the cold-pressed powders are shown in table I.

Sintering of powder mixtures. - The cold-pressed rods were further consolidated by sintering. The standard sintering treatment was conducted in an all-metal resistively heated furnace and consisted of (1) heating to 1500° C in Ar for 2 hours, (2) adding N_2 to the Ar and holding at 1500° C for 1/2 hour, (3) heating to the sintering temperature in 1 hour, (4) holding at the sintering temperature for 3 to 10 hours, (5) cooling to 1500° C in about 10 minutes, (6) removing N_2 from the atmosphere and holding at 1500° C for 1/2 hour, and (7) cooling to less than 200° C in about 15 minutes. The Ar and Ar-N_2 gas mixtures contained less than 10 ppm O_2 or water and were continuously regulated at a pressure of 1 atmosphere and a flow rate of 15 standard cubic feet per hour (1.2×10^{-4} m^3/sec). Nitrogen was added to the Ar at temperatures over 1500° C to prevent decomposition of UN. Nitrogen was not used below 1500° C to prevent formation of uranium sesquinitride (U_2N_3).

Pneumatic impaction of sintered cermets. - A limited study also was conducted to establish the feasibility of further consolidating sintered cermets by hot pneumatic im-

TABLE I. - PROPERTIES OF URANIUM NITRIDE, TUNGSTEN, AND MOLYB-

DENUM POWDER BEFORE AND AFTER MIXING AND

COLD PRESSING (70 000 psi (500 MN/m^2))

(a) Before mixing and pressing

Powder lot number[a]	Particle surface area,[b] m^2/g	Particle size,[c] μm	Chemical analysis, ppm								
			N/U[d]	O	C	Si	Ca	Cr	Fe	Ni	Mo
UN-1	0.18	1.6	1.01	1250	257	20	<5	<10	330	350	90
UN-2	1.07	.26	.99	3825	549	15	30	200	300	350	15
W-1[e]	.16	4.5	ND[f]	250	11	<1	<1	3	2	5	10
W-2[e]	1.14	.88	ND	1255	37	2	<1	5	15	10	50
Mo-1[e]	.34	4.5	ND	53	54	20	5	5	30	ND	ND

(b) After mixing and pressing

Cermet lot number	Uranium nitride lot number	Metal lot number	Fuel (UN) loading, vol. %
A	UN-1	W-1	44
B	UN-1	W-2	50
C	UN-2	W-2	56
D	UN-1	Mo-1	48
E	UN-2	Mo-1	49

[a]UN, W, and Mo denote the chemical symbols of each powder lot.
[b]BET analysis.
[c]Particle sizes of both UN lots were calculated from surface area measurements assuming spherical particles; particle sizes of W and Mo lots were measured by a Fisher subsieve analyzer.
[d]Stoichiometry ratios were calculated by assuming all O_2 exists in the form of UO_2.
[e]Hydrogen "cleaned" for 18 hr at 800° C.
[f]Not determined.

paction. A cermet previously sintered for 3 hours in Ar (33 vol. %) - N_2 (67 vol. %) at 2200° C was machined to fit into a Mo can. The can was sealed by electron beam welding and then pneumatically impacted at 2050° C and 28 000 foot-pounds (38 000 joules).

Density of Sintered Cermets

The densities of sintered cermets are presented in table II. Most of the density measurements (water immersion were made on single specimens. However, several of the density values shown in table II pepsent the average of 2 to 3 measurements, the multiple measurements generally agreed within 2 percentage points.

TABLE II. - PROPERTIES OF PRESSED SINTERED TUNGSTEN - URANIUM

NITRIDE AND MOLYBDENUM - URANIUM NITRIDE CERMETS

Cermet lot (type)	Specimen number	Sintering treatment			Density,[a] percent of theoretical	Chemical analysis		
		Temperature, $^\circ$C	Time, hr	Atmosphere (N$_2$ in Ar), vol. %		N/U[b]	O, ppm	C, ppm
A (W-UN)	1	2200	10	67	82	1.04	560	23
	2	↓	5	67	82	1.02	800	5
	3		3	67	83	.98	1195	ND[c]
	4	↓	3	0	86	.99	1485	ND
B (W-UN)	1	1500	8	0	81	1.02	1009	ND
	2	2200	10	67	92	1.00	1290	ND
	3	↓	5	67	90	1.02	1137	11
	4		3	67	86	1.04	1290	54
	5	↓	3	10	88	1.03	1167	76
	6	↓	3	0	97	[d].96	1855	241
	7	2500	5	67	95	.99	1930	ND
C (W-UN)	1	2200	3	67	95	0.96	2673	ND
	2	2200	3	0	95	.97	3740	ND
D (Mo-UN)	1	2000	5	67	83	1.02	1135	ND
	2	2200	5	67	89	1.03	1135	52
	3	↓	3	67	87	.97	1195	52
	4		3	10	89	1.02	1198	112
	5	↓	3	0	98	.98	1935	ND
E (Mo-UN)	1	2200	3	67	98	0.99	2990	ND
	2	2200	3	0	98	[e].97	3933	ND

[a]Determined by water immersion of specimens sealed on their external surfaces
   with lacquer.
[b]Stoichiometry ratios were calculated by assuming all O$_2$ exists in the form of UO$_2$;
   precision of N/U = ±4 percent of value shown.
[c]Not determined.
[d]Free U in grain boundaries.
[e]Mo-U phase formed.

Several variables including powder particle size and sintering temperature, time, and atmosphere affected the density of cermets. In fact, cermet density could be controlled by proper selection of these variables. Although the effects of UN stoichiometry and O_2 impurity level (see table II) on density also were examined, no correlation could be made. However, the UN stoichiometry and/or O_2 level could have affected the results of the other variables. The following sections describe the effect of these variables on density.

Powder particle size. - One method commonly used to control the sintered density of pressed powders is to vary the powder particle size, that is, the particle surface area. In this study, the particle surface area of both the UN and the W were varied. Figure 1 shows the effect of varying the powder surface area on the density of cermets which were
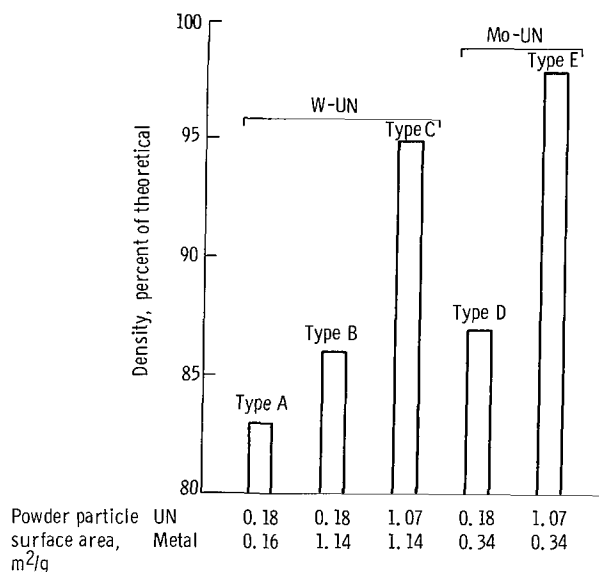

Figure 1. - Effect of powder surface area on density of W - 50-volume-percent UN and Mo - 50-volume-percent UN cermets sintered for 3 hours at 2200° C in an Ar (33 vol. %) - N_2 (67 vol. %) atmosphere.

sintered for 3 hours at 2200° C in an Ar (33 vol. %) - N_2 (67 vol. %) atmosphere. Increasing the W powder surface area resulted in only a slight increase in cermet density (compare types A and B). However, by also increasing the UN powder surface area (type C), a considerably larger increase in the cermet density was achieved. Likewise, a larger increase in the density of Mo-UN cermets was achieved by increasing the UN powder surface area (compare types D and E). The finer particles (larger surface area) apparently resulted in finer pores which required less volume diffusion (and time) in order to densify the cermets.
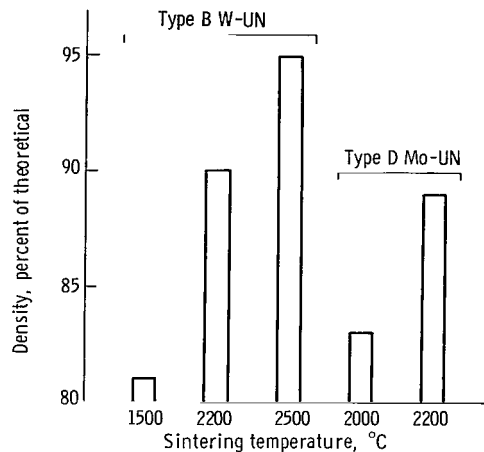
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Figure 2. – Effect of sintering temperature on density of cermets sintered for 5 hours (except for the type B specimen sintered for 8 hr at 1500° C) in an Ar (33 vol. %) – N_2 (67 vol. %) atmosphere.
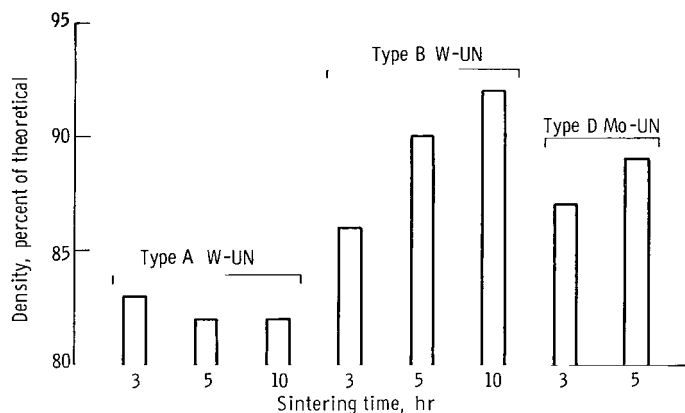

Figure 3. – Effect of sintering time on density of cermets sintered at 2200° C in an Ar (33 vol. %) – N_2 (67 vol. %) atmosphere.

Sintering temperature. – The effect of sintering temperature on the density of type B and D cermets is shown in figure 2. Increasing the sintering temperature resulted in increased cermet densities. For example, increasing the temperature from 1500° to 2500° C resulted in an increase from 81 to 95 percent of theoretical density for type B W-UN cermets. Similarly, type D Mo-UN cermets exhibited an increase in density with temperature.

Sintering time. – The effect of increasing sintering time at 2200° C from 3 to 10 hours is illustrated in figure 3. Time at temperature had little, if any, effect on density of type A W-UN cermets or type D Mo-UN cermets. However, type B W-UN cermets (which were prepared from a finer W powder than type A cermets) exhibited an increase in density of 4 percentage points when the sintering time was increased from

3 to 5 hours. A further increase in time to 10 hours resulted in a further density increase of only 2 percentage points. Apparently, the sintering time (in excess of 3 hr) is effective in varying cermet densities only when the powder particle sizes are sufficiently small.

Sintering atmosphere. - Unless a sufficient N_2 pressure is present at elevated temperatures, UN will decompose into U and N_2 according to thermodynamic and phase diagram studies (refs. 4 and 10). For example, a N_2 pressure of 8×10^{-4} atmosphere at 2200° C and 3×10^{-2} atmosphere at 2500° C is required to prevent decomposition. The following paragraphs describe the effect of varying the N_2 content of the sintering atmosphere on the density of cermets.

All five types of cermets were sintered for 3 hours at 2200° C in atmospheres of Ar containing 0 to 67 volume percent of N_2. The effects of these sintering treatments on cermet density are presented in figure 4. For W-UN cermets, complete removal of N_2
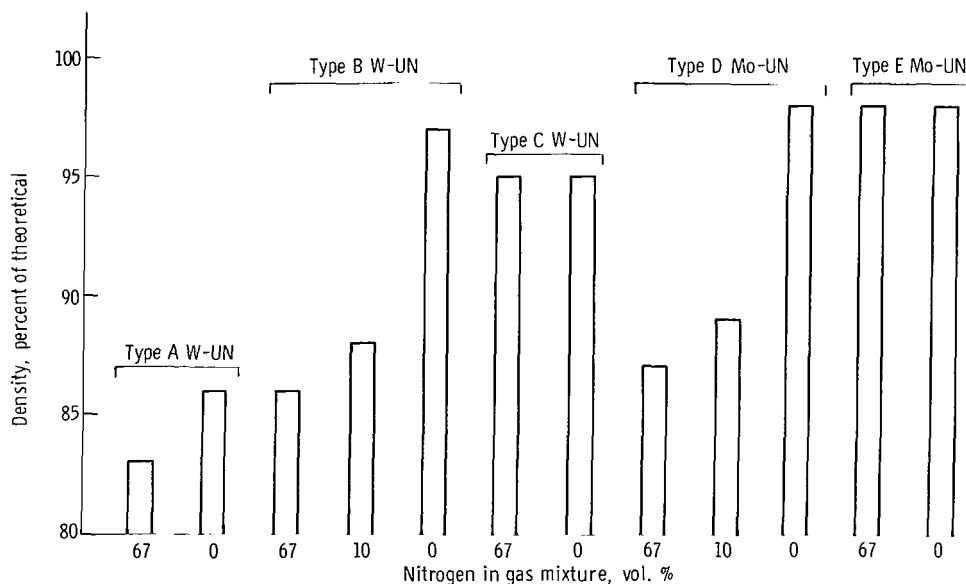

Figure 4. - Effect of sintering atmosphere on density of W-UN and Mo-UN cermets sintered for 3 hours at 2200° C in designated Ar-N_2 gas mixtures.

from the atmosphere resulted in a relatively large density increase in type B cermets, a moderate increase in type A cermets, and no change in type C cermets. The larger density increase in type B than in type A cermets apparently resulted from the finer W particles in type B cermets. The very fine powder particle size in type C cermets permitted relatively high densities to be achieved even in the presence of N_2. Therefore, the effect of removing N_2 on cermet density was obscured.

The effect of removing N_2 from the sintering atmosphere on cermet density is shown

in figure 4 for type B W-UN cermets. Lowering the N_2 content from 67 to 10 volume percent of the atmosphere had little effect on the cermet density. It is believed that the sintering N_2 pressure could be reduced to the N_2 pressure at which UN decomposes before U formation and liquid-phase sintering would occur.

For Mo-UN cermets, the changes in cermet densities with N_2 content in the atmosphere were similar to the W-UN cermets with similar UN particle sizes. For example, type D cermets exhibited no significant increase in density as N_2 pressure was decreased from 67 to 10 volume percent of the atmosphere. But when N_2 was completely removed from the sintering atmosphere, the density greatly increased. In the case of type E cermets containing the finer UN powder, high densities were achieved even when N_2 was present during sintering. Thus, no significant increase in density was measured on these cermets when sintered without N_2.

Chemical Compatibility of Cermets

The purpose of this section is to characterize the compatibility between cermet components and also to comment on reasons for associated changes in cermet density. Briefly, UN has generally proved to be compatible with both W and Mo at temperatures up to 2200° C provided that N_2 is present in the atmosphere. Different levels of O_2 and carbon (C) impurities in the starting powders (table I) had no apparent effect on compatibility. The effect of initial UN stoichiometry on compatibility was not determined because of the similar stoichiometry (N/U) of the UN starting powders (see the appendix for the significance of chemical analysis).

The sintering atmosphere conditions appeared to have the major effect on compatibility of cermets. For example, when N_2 was not present at temperatures over 1500° C, UN decomposed to form U which then reacted with the matrix metal. This reaction appeared to cause liquid-phase sintering and tended to accelerate the densification of cermets. The following paragraphs summarize the effects of the sintering atmosphere on the compatibility of UN with W and Mo.

Tungsten - uranium nitride cermets. - Neither metallographic examination (fig. 5(a)) nor chemical analysis (table II) of type A cermets sintered with or without N_2 revealed any UN decomposition or reaction with W. Although some indication of UN decomposition (e.g., the appearance of free U or liquid-phase sintering in the microstructure) was expected in the cermet sintered without N_2, evidence of decomposition may have been obscured by the relatively large particles and voids. For example, U could have been present in undetectable amounts on void surfaces. In addition, the sintering time may not have been long enough to permit appreciable densification of the relatively large particles by liquid-phase sintering.
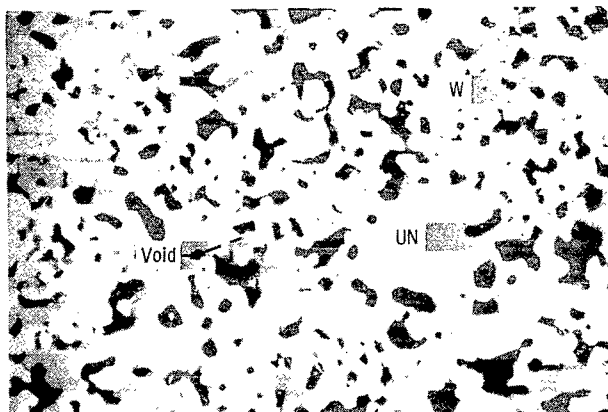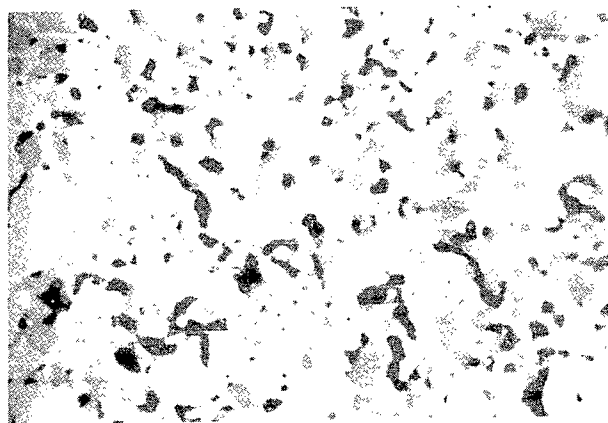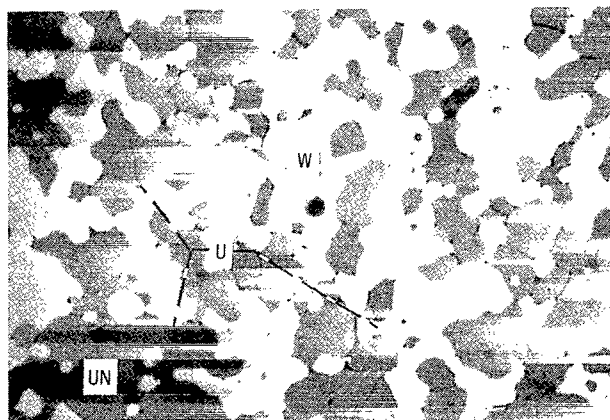
(a-1) Density, 83 percent of theoretical.

(a-2) Density, 86 percent of theoretical.

(a) Type A; particle surface area: UN, 0.18 square meter per gram; W, 0.16 square meter per gram.
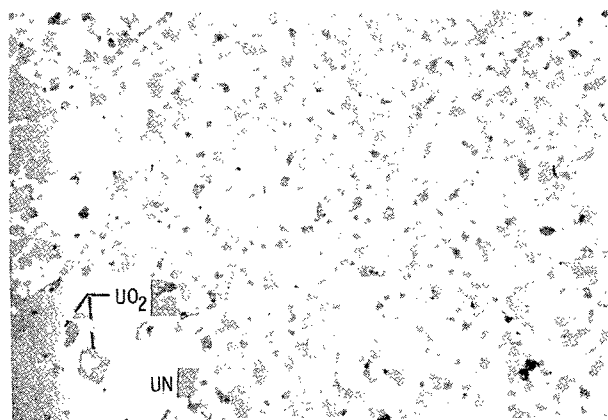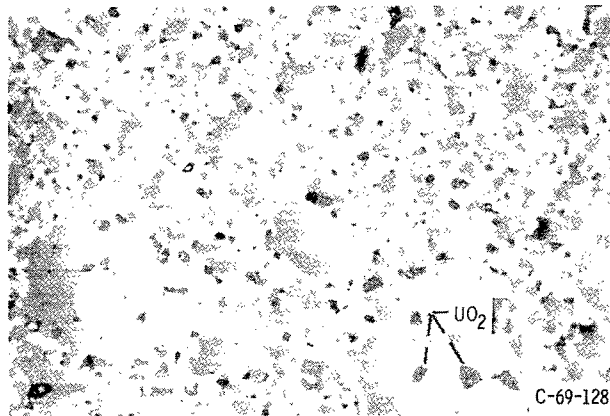

(b-1) Density, 86 percent of theoretical.

(b-2) Density, 97 percent of theoretical.

(b) Type B; particle surface area: UN, 0.18 square meter per gram; W, 1.14 square meters per gram.

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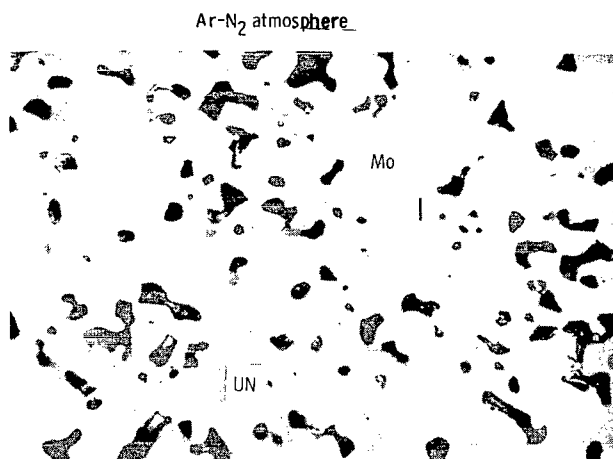(c-1) Density, 95 percent of theoretical.

(c-2) Density, 95 percent of theoretical.

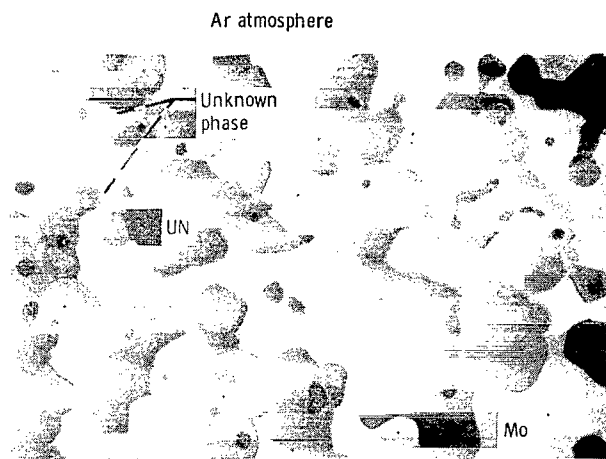(c) Type C; particle surface area: UN, 1.07 square meters per gram; W, 1.14 square meters per gram.

Figure 5. - Microstructures of cermets sintered for 3 hours at 2200° C in Ar (33 vol. %) - N$_2$ (67 vol. %) and Ar atmospheres. X500.

In figure 5(b), the microstructure of a type B cermet sintered in an Ar-N_2 mixture shows less porosity than that observed in type A cermets (which were produced from a coarser W powder). When type B cermets were sintered without N_2, the porosity was nearly eliminated. In addition, many of the UN dispersoids became more rounded and a third phase developed in the UN grain boundaries. This phase tarnished when heated indicating that it was U. Chemical analysis (table II) also indicated substoichiometric UN.

The high density achieved in the type B cermet sintered without N_2 probably resulted from liquid-phase sintering. At elevated temperatures, the UN decomposed into molten U and N_2. The slight mutual solubility between W and U at elevated temperatures (ref. 11) then permitted liquid-phase sintering and densification to occur.

Ar-N_2 atmosphere

Ar atmosphere

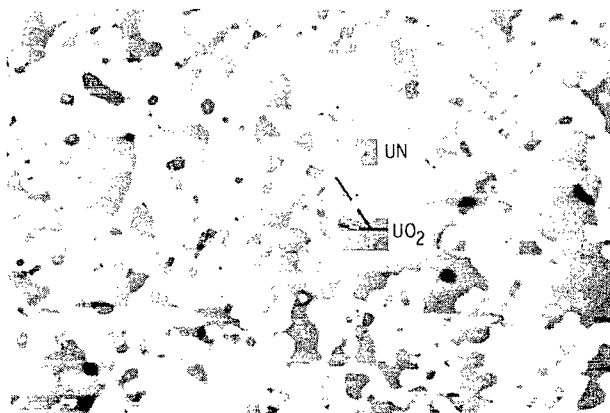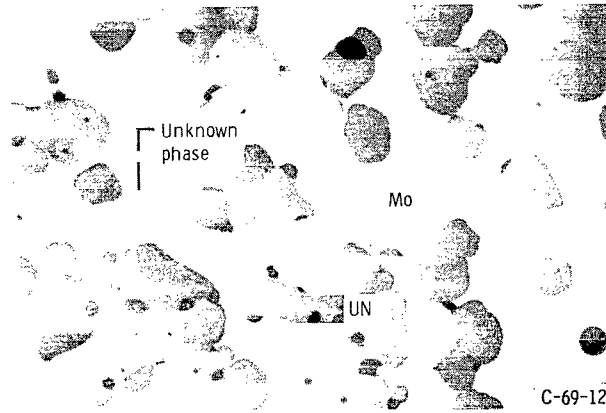(d-1) Density, 87 percent of theoretical.

(d-2) Density, 99 percent of theoretical.

(d) Type D; particle surface area: UN, 0.18 square meter per gram; Mo, 0.34 square meter per gram.

(e-1) Density, 98 percent of theoretical.

(e-2) Density, 98 percent of theoretical.

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(e) Type E; particle surface area: UN, 1.07 square meters per gram; Mo, 0.34 square meter per gram.

Figure 5. - Concluded.

Type C cermets achieved very high densities when sintered with or without N_2 (fig. 5(c)). The high densities were achieved without the formation of U detectable by metallography. The reasons why U was not observed in the cermets sintered without N_2 (as would be expected from UN decomposition) are not fully understood. Perhaps the small UN dispersoid size provided enough surface area to prevent accumulation of observable amounts of U in any one area.

The microstructure (fig. 5(c)) also illustrates the relatively high UO_2 content and the fine pore and UN dispersoid size, typical of type C cermets. Although no special attempt was made in this study to prevent agglomeration of these fine UN dispersoids, it is clear that additional work is needed to attain an even dispersion of fine UN dispersoids in a fuel element.

Molybdenum - uranium nitride cermets. - Figure 5(d) shows the effect of N_2 in the sintering atmosphere on type D cermets. These Mo-UN cermets sintered in N_2 exhibited an even dispersion of irregular pores (adjacent to UN dispersoids) similar to the W-UN cermets. However, when these cermets were sintered without N_2, they achieved nearly theoretical density. Also, another phase appeared in the microstructure adjacent to UN dispersoids (fig. 5(d)). Electron microprobe analysis identified this phase as approximately 75 percent Mo, 25 percent U, and possibly 1 to 2 percent N_2. (The N_2 analysis is questionable because of U interference.) The U in this phase probably originated from decomposed UN which then dissolved Mo and caused liquid-phase sintering. The rounded UN dispersoids are also indicative of liquid-phase sintering. The most likely reason for the more extensive liquid-phase sintering in Mo-UN than in W-UN cermets is the greater high temperature solubility between Mo and U than between W and U. For example, U dissolves 16 weight percent of Mo and 1.5 weight percent of W at 1500° C (ref. 11).

Microstructures of type E cermets are shown in figure 5(e). Similar to type C W-UN cermets, the finer UN particle size permitted relatively high densities to be achieved even in a N_2 sintering atmosphere. The type E cermet sintered without N_2 appeared to be similar to the type D specimen sintered without N_2.

Size and Distribution of Pores in Cermets

In addition to characterizing porosity, as shown in microstructures, selected specimens were analyzed with a mercury porosimeter. This analysis made possible determination of the mean pore diameter, the pore size distribution, and the percent of open porosity (i.e., the percent of pore volume which is connected to external surfaces). The results of types A, B, and D cermets (after sintering for 3 hr at 2200° C in an Ar (33 vol. %) - N_2 (67 vol. %) atmosphere) are presented in table III and are summarized in the following paragraph.

TABLE III. - POROSITY MEASUREMENTS OF CERMETS

SINTERED FOR 3 HOURS AT 2200° C IN ARGON

(33 VOL. %) - NITROGEN (67 VOL. %)

ATMOSPHERE

Cermet lot (type)	A (W-UN)	B (W-UN)	D (Mo-UN)
Density,[a] percent of theoretical	83	86	88
Open porosity, percent of total porosity connected to cermet surfaces[b]	100	80	90
Average pore diameter,[b] μm	1.70	1.14	1.72
Pore size distribution, vol. % of pores in given size ranges[b]			
Pore size, μm:			
>5	0	0	0
5 to 4	.8	0	0
4 to 3	1.4	1.4	0
3 to 2	0	1.4	2.0
2 to 1	94	65	95
1 to 0.7	2.2	21.5	2.0
0.7 to 0.5	.8	6.2	0
0.5 to 0.3	.8	1.4	1.0
0.3 to 0.1	0	3.1	0
<0.1	0	0	0

[a]Determined by water immersion method.
[b]Determined by intrusion of pressurized mercury into pores.
   Pores ranging from 0.035 to 100 μm in diameter could be
   measured by this method.

From 90 to 100 percent of the total pore volume in type A and D cermets was connected to the specimen surfaces, and 1- to 2-micrometer-diameter pores accounted for about 95 percent of this open pore volume. Type B cermets were fabricated from finer W powder than type A cermets. As a result, type B cermets exhibited less open porosity (about 80 percent of the total pore volume) and a finer open pore size (0.1- to 1-μm-diameter pores accounted for 32 percent of the open pore volume) than type A cermets.

The implications of the open porosity measurements in cermets are important for potential reactor applications. For if such cermets are used in a reactor, fission gases released into internal pores could easily be vented from the cermets. The "venting efficiency" (i.e., open porosity) appears to be better in cermets fabricated from powders

with larger particle sizes. Such venting requires, however, that the pores are stable and that their access to the external surfaces is not blocked during reactor operation.

Thermal Stability of Tungsten – Uranium Nitride and Molybdenum – Uranium Nitride Cermets

Some of the sintered cermets were thermally treated (3 hr at 2200° C) in the absence of N_2 to get an indication of the chemical and pore stability in the cermets. The results of the stability tests on type A, B, and D cermets are presented in figure 6. Both W-UN cermets (types A and B) exhibited a 5- to 6-percent increase in density (i.e., about one-third of the open porosity was lost). Also, the UN became slightly substoichiometric as a result of this test. The UN in the type D Mo-UN cermet lost a larger amount of N_2 and became more substoichiometric than the W-UN cermets. As may be seen in the photomicrograph in figure 6(c), a second phase (similar to that observed in figs. 5(d) and (e)) was formed. This phase was apparently liquid at 2200° C and caused liquid-phase sintering and cermet densification to occur.

Thus, it appears that W-UN cermets offer more thermal stability than Mo-UN cermets in the absence of N_2. This stability may be quite important when selecting the best cermet metal for use in reactor applications where N_2 may not be present.

Hot Pneumatically Impacted Cermets

Attempts to achieve theoretical density in W-UN cermets by sintering were not successful except when either (1) the UN decomposed and liquid-phase sintering occurred or (2) very fine UN particles (which, because of their fineness, were highly reactive and contained considerable oxygen impurity) were used. Therefore, further densification of high density W-UN cermets (type B) was attempted by a method which would not require liquid-phase sintering or the use of fine UN particles.

Hot pneumatic impaction of a sintered type B W-UN cermet resulted in an increase in cermet density from 86 to 96 percent of theoretical. The microstructure of the impacted cermet is illustrated in figure 7. In addition to the high density, photomicrographs reveal two other interesting phenomena. First, a small amount of UN was found in the Mo container can. Although the reason for this unknown, it appears that some chemical reaction caused the migration of UN into the Mo grain boundaries. Second, UO_2 impurities were observed in the UN along the UN-Mo can interface. This UO_2 may have originated before impaction at localized hot spots which developed when the cermet

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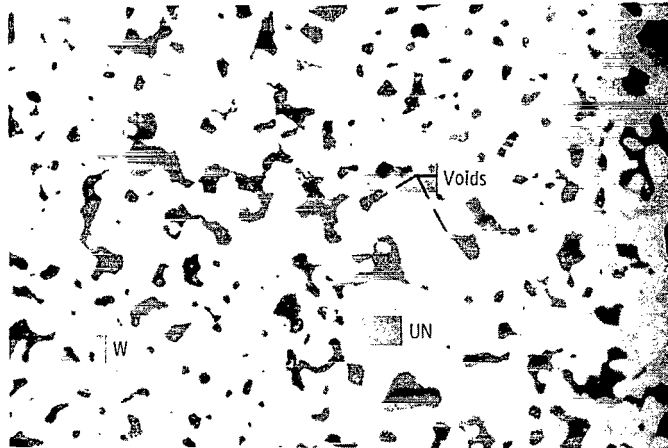
(a) Type A; W-UN; theoretical density, 83 to 88 percent; N/U, 0.98 to 0.97.
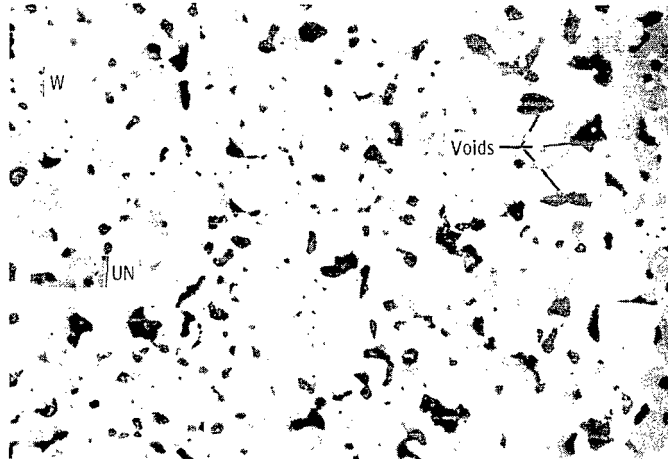
(b) Type B; W-UN; theoretical density, 86 to 92 percent; N/U, 1.04 to 1.01
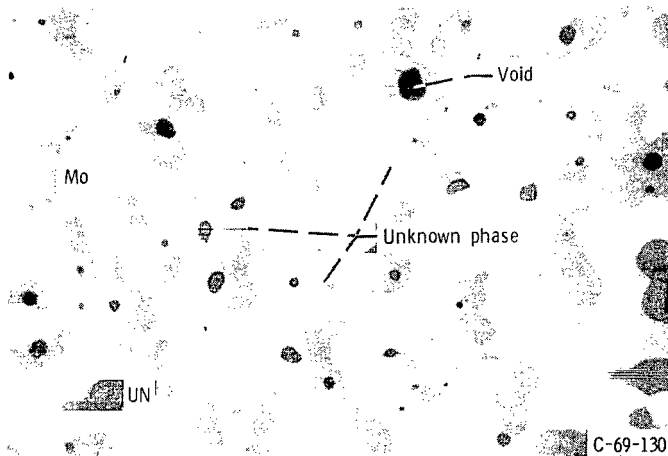
(c) Type D; Mo-UN; theoretical density, 89 to 98 percent; N/U, 1.02 to 0.94.

Figure 6. - Effect of 3-hour 2200° C heat treatment (in Ar) on microstructure, density, and UN stoichiometry (N/U) of sintered cermets. X500.
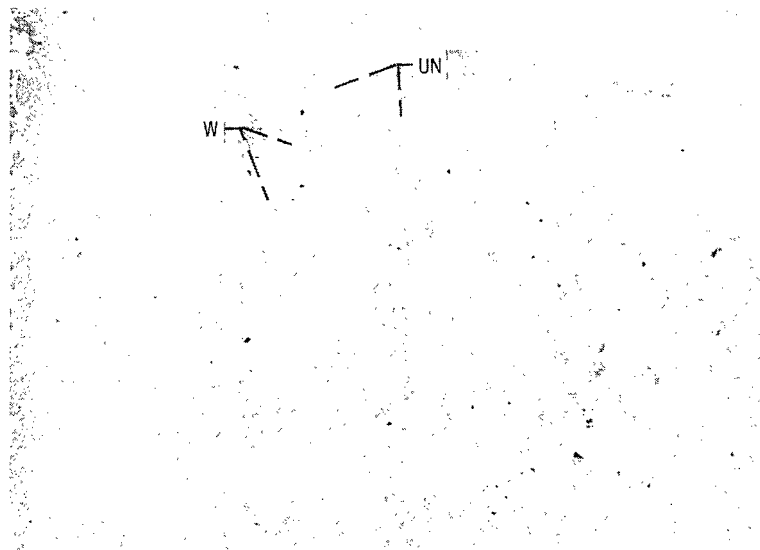
(a) W-UN cermet. X100.
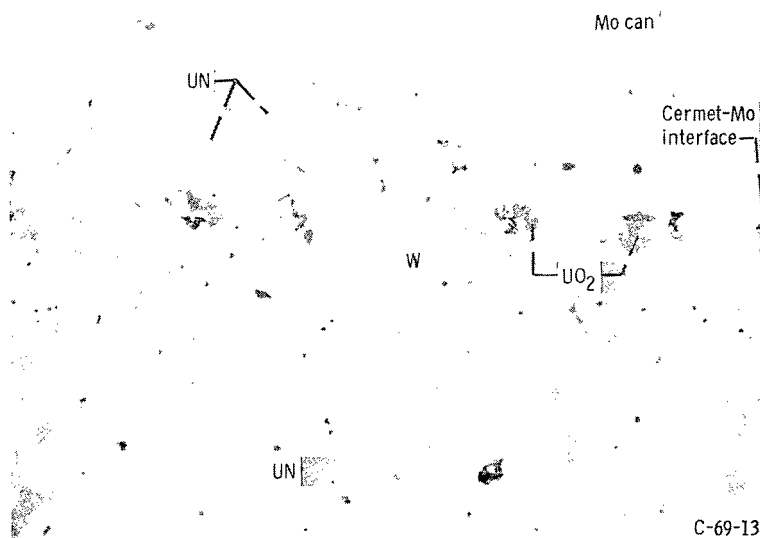
(b) W-UN cermet. X500.

Figure 7. – Type B; W-UN cermet consolidated by hot pneumatic impaction. This specimen exhibited 98 percent of theoretical density, nitrogen to uranium ratio of 1.04, and 1065 ppm of oxygen.

was machined (in air) to fit into the Mo can. It would be relatively easy to remove this UO_2 by machining (in an inert atmosphere if necessary).

This experiment demonstrated the feasibility of producing highly dense cermets from powder mixtures without either decomposing or oxidizing UN. Where high density cermets are required, hot pneumatic impaction of presintered cermets offers a definite advantage over cold-pressing and sintering as a method of powder consolidation.

COATED-PARTICLE CERMETS

It is necessary to coat UN particles with a metal prior to consolidation in order to produce cermets (containing over about 50 vol. % of UN) which exhibit a continuous metal matrix and complete separation of the UN particles. Such an approach was successfully demonstrated for $W-UO_2$ cermets containing up to 70 volume percent of UO_2 (ref. 12). In this study, relatively large UN particles (75 to 150 μm diameter) were spheroidized in a plasma torch and then coated with W or Mo by an electron beam vaporization process to obtain metal-coated particles containing 50 to 70 volume percent of UN. The coated particles were consolidated into cermets by hot isostatic compaction.

Fabrication of Coated-Particle Cermets

Preparation of spherical uranium nitride particles. - Two lots of spherical UN particles (designated as lots X and Y) were prepared by two suppliers under development contracts with NASA. Both suppliers spherodized the particles by similar plasma torch processes. The following paragraphs described the process and compare the powder lots.

Irregular-shaped, hyperstoichiometric UN particles were first prepared by nitriding U chips at 850° C. These particles were treated at 1400° C in vacuum to attain the stoichiometric composition. After classifying to obtain the desired particle size ranges, the particles were fed into a N_2 atmosphere plasma torch. The resultant particles were spherical and consisted of a UN surface case and a UN dendrite structure in a U matrix. These particles were then subjected to the following equilibration heat treatments in N_2 at the specified pressures to convert the U to UN while avoiding formation of U_2N_3:

Lot X: 15 hours at 1100° C and 0.020 atmosphere of N_2 plus 80 hours at 1400° C and 0.53 atmosphere of N_2

Lot Y: 8 hours at 1100° C and 0.013 atmosphere of N_2 plus 10 hours at 1350° C and 0.72 atmosphere of N_2 plus 2 hours at 1350° C in vacuum

TABLE IV. - PROPERTIES OF URANIUM NITRIDE SPHERICAL PARTICLES

UN lot number[a]	X1	X2	X3	Y1	Y2
Particle size range, μm	100 to 150	75 to 100	100 to 150	40 to 75	75 to 150
Chemical analysis, ppm:					
N/U[b]	1.01	1.00		0.97	
O	640	746		2980	
C	170	180		339	
H	1	2		ND[c]	
Al	10	ND		20	
B	1	0.6		.5	
Be	ND	10		1	
Ca	1	2		25	
Cu	10	10		6	
Fe	33	50		60	
Mg	2	2		10	
Mo	10	20		25	
Ni	7	10		10	
Si	175	500		100	

[a]X and Y denote the two UN vendors.

[b]Stoichiometry ratios were calculated by assuming all O_2 exists in the form of UO_2.

[c]Not determined.

Each lot was then classified into the desired particle size ranges. Yields (based on weight) of acceptable particles ranged from 10 to 30 percent of the starting UN powder.

The properties of the two spherical UN powder lots are compared in table IV and in figure 8. Oxygen impurity was considerably lower in lot X than in lot Y powder indicating that the former lot was processed in a more leak-tight system. The UN stoichiometry was quite good in lot X, as evidenced by both chemical analysis and the lack of much U or U_2N_3 in its microstructure (figs. 8(a) to (d)). Lot Y, on the other hand, exhibited evidence of substoichiometry by both the chemical analysis and by the U present in the microstructure (figs. 8(e) and (f)). Apparently, the final heat treatment on lot Y powder was insufficient to produce stoichiometric UN.

The densities of the two types of powder also may be compared. Considerable difficulty was encountered in producing uniformly dense particles in lot X, while lot Y particles were generally quite dense. The internal pores found in the particles (e.g., fig. 8(d)) are believed to have resulted from the loss of liquid U (which melts at 1130° C) from particles before the U was nitrided in the equilibration heat treatment. It is theorized that a UN case must be formed around each particle during plasma spheroidization to prevent such loss. The supplier of lot X particles apparently could not control the
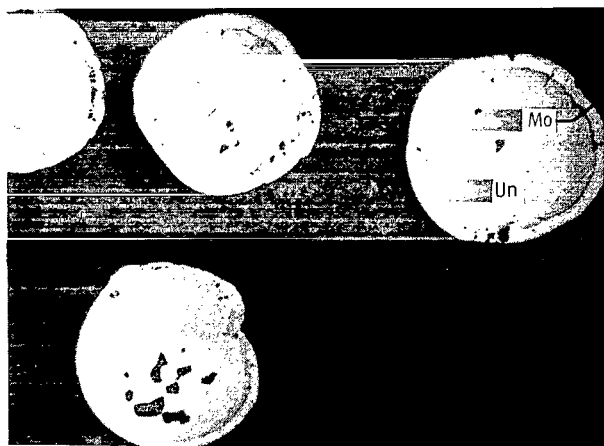
(a) Lot number, X2M; Mo - 60-volume-percent UN; particle diameter, 70 to 100 micrometers.
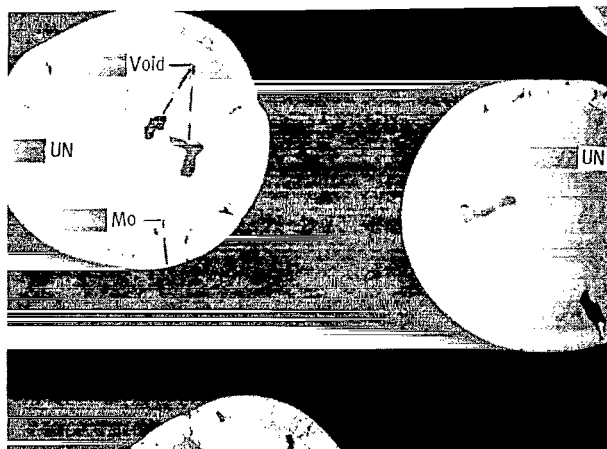
(b) Lot number, X1M; Mo - 70-volume-percent UN; particle diameter, 100 to 150 micrometers.
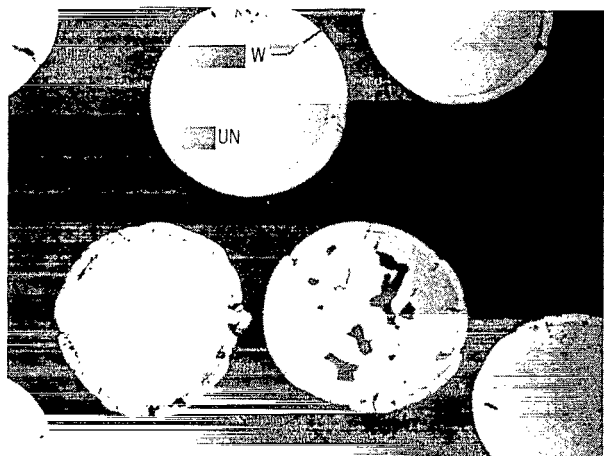
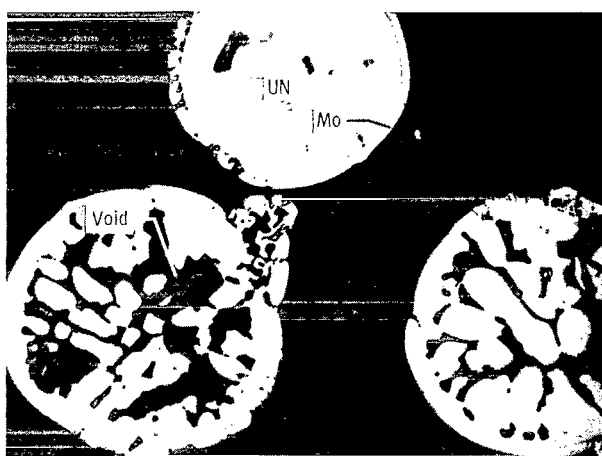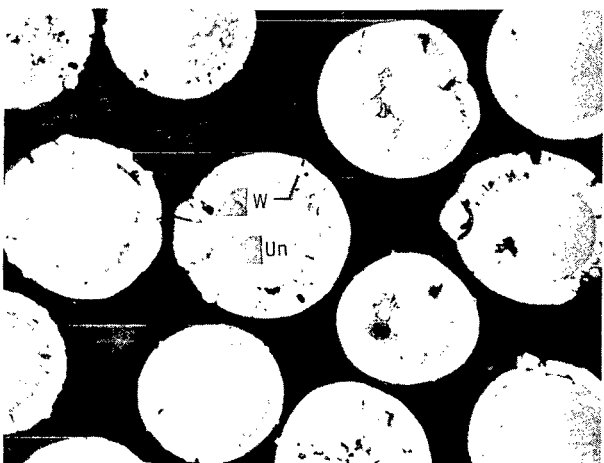(c) Lot number, X2W; W - 70-volume-percent UN; particle diameter, 75 to 100 micrometers.

(d) Lot number, X3M; Mo - 70-volume-percent UN; particle diameter, 100 to 150 micrometers.

(e) Lot number, Y1W; W - 50-volume-percent UN; particle diameter, 40 to 75 micrometers.

(f) Lot number, Y2W2; W - 60-volume-percent UN; particle diameter, 75 to 150 micrometers.

Figure 8. - Photomicrographs of metal-coated UN particles. X250.

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plasma arc conditions as effectively as did the supplier of lot Y particles to produce such a case consistently. Attempts to produce a UN case around particles after spherodication by nitriding at temperatures below the melting point of U were only partly successful.

Coating of uranium nitride particles. - The spherical UN particles were coated with W or Mo by one of the UN vendors. The process of H_2 reduction of gaseous metal halides could not be used to coat the particles because of reactions between the halides and UN. Therefore, the particles were coated by a process in which a cage of tumbling UN particles was placed in the path of vaporized W or Mo. Feed rods of these metals were vaporized by electron beam heating. This proprietary process had previously been developed by one of the UN vendors for other materials.

The fuel (UN) loading and impurity level of each lot of coated UN particles prepared for this study are given in table V. Based on analysis of particles before and after coating, the coating process appears to have introduced some O_2 and H_2 impurities into the particles. These impurities probably originated from leaks in the coating apparatus through which O_2 and water vapor could enter the apparatus. In general, the fuel loadings attained were within 2 to 3 percentage points of the desired values.

Typical microstructures of Mo-coated particles are shown in figures 8(a) and (b); W-coated particles are shown in figures 8(c) to (f). Coated particles with fuel loadings ranging from 50 to 70 volume percent and UN particle sizes ranging from 40 to 150 micrometers in diameter are illustrated. Observe that the coatings were condensed on external particle surfaces, but not in internal pores connected to the particle surfaces.

Pores and microcracks were present in most of the metal coatings. Acid leach tests (the acid attacked UN but not Mo or W) indicated that more than 75 percent of the coatings. were discontinuous (table V). The coating discontinuities (e.g., cracks) are believed to have formed when the particles were cooled after coating and to have resulted from the difference in thermal expansivity between UN and W or Mo.

Consolidation of coated uranium nitride particles. - The coated UN particles were loaded into Mo cans and vibratorily compacted under an Ar atmosphere. The Mo can was then sealed under vacuum by electron beam welding and inspected for leaks. Finally, the cans were hot isostatically compacted for 3 hours in helium at 1650° C and 30 000 psi (200 MN/m^2).

Characterization of Coated-Particle Cermets

Duplicate coated-particle-cermet specimens were evaluated by both density measurements and by metallography. The following sections summarize the results of these evaluations.

TABLE V. - PROPERTIES OF COATED URANIUM NITRIDE SPHERICAL PARTICLES

Metal coating	W							Mo		
Coated particle lot number[a]	X1W	X2W	X3W	Y1W	Y2W1	Y2W2	Y2W3	X1M	X2M	X3M
Fuel (UN) loading, vol. %	71	70	68	47	48	62	68	71	65	68
Fuel (UN) particle diameter size range, μm	100 to 150	75 to 100	100 to 150	40 to 75	75 to 150	75 to 150	75 to 150	100 to 150	75 to 100	100 to 150
Coating integrity test, percent UN leached out of coated particles by acid	ND[b]	ND	ND	99	93	77	96	ND	ND	ND
Chemical analysis, ppm:										
O	1890	980	1320	3800	3800	1740	2700	1190	1690	1740
C	110	82	134	126	136	180	194	140	114	170
H	75	47	40	120	88	24	52	51	76	64
F	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Cl	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
S	<10	14	12	<10	<10	<10	<10	<10	<10	<10
Al	<25	<25	<25	<20	<25	<50	<25	<20	<20	<20
Ca	<50	<50	<50	ND	ND	ND	ND	<50	<50	<50
Co	<25	<25	<25	<25	<25	<10	<25	<5	<5	<5
Cr	<25	<25	<25	<25	<25	40	<25	<10	<10	<10
Cu	<25	<25	<25	25	<25	<20	<25	10	<6	10
Fe	50	75	75	50	75	100	100	75	50	50
Mg	<25	<25	<25	<25	<25	<20	<25	<10	<10	<10
Mn	<25	<25	<25	<25	<25	<20	<25	<10	<10	<10
Mo	ND	ND	ND	200	200	200	200	ND	ND	ND
Ni	<25	<25	<25	<25	<25	20	30	<10	<10	<10
Si	<50	<50	<50	100	100	150	100	<50	<50	<50
Sn	<20	<20	<20	<25	<25	<10	<25	<20	<20	<20
Ti	<25	<25	<25	<25	<25	<20	<25	10	10	10
V	<25	<25	<25	<5	<25	<5	<25	<5	<5	<5
Zr	<50	<50	<50	<50	<50	<50	<50	<25	<25	<25
Cermet density, percent of theoretical after hot isostatic consolidation[c]	100	(d)	99	(d)	97	99	98	100	99	100

[a]First letter and number denote UN particle lot; last letter denotes metal coating.
[b]Not determined.
[c]"Pack" density of particles prior to consolidation was 55 to 60 percent of theoretical.
[d]Consolidation was not achieved because of leaks in canning material.

Density of cermets. - The density of all the coated-particle cermets was at least 97 percent of theoretical according to water immersion measurements (table V). These density values were confirmed by the cermet microstructures (figs. 9 and 10). Note that most of the pores within the UN particles and all the metal-coating cracks originally in the coated particles (fig. 8) disappeared after hot pressing. The few pores observed in these cermets were usually located either in the UN dispersoids or at interstitial positions between the coated particles (fig. 9(c)). The pores within the UN originated during particle spheroidization (fig. 8) and the pores within the matrix resulted from incomplete consolidation. Apparently, slightly higher hot pressing temperatures, times, and/or pressures would be required to eliminate porosity completely in all cermets.

Although pores in the UN particles were not completely eliminated by hot pressing, the initial UN particle density had little effect on the cermet density. For example, two lots of UN powder (X3W and X3M) exhibited a substantial amount of internal porosity in the UN (fig. 8(d)). One of these lots (X3W) was not densified because of leaks which developed in the Mo containment can. However, the other lot (X3M) was consolidated to theoretical density. The microstructure of this cermet (fig. 10(e)) reveals the distortion of the formerly spherical particles as a result of densification. Based on these results, it is apparent that UN is plastic enough at 1650° C to be deformed by 30 000 psi (200 MN/m^2). Any attempt to retain porosity in UN would have to be done at lower temperatures and/or pressures.

No attempt was made to vary the density of coated-particle cermets. However, the density of these cermets probably could be controlled easily by varying the hot pressing temperature, pressure, and time. This method of density control would affect the number and size of pores in the metal matrix. In addition, the hot pressing conditions would probably affect the extent to which cracks in the particle metal coatings are healed. It is also conceivable that the internal UN porosity could be controlled if better control could be gained over the plasma spheroidization process. Thus, it is possible that vents (i.e., internal, interconnected voids) could be incorporated into coated-particle cermets to allow escape of fission gases from the UN dispersoids (by internal dispersoid pores), through the metal coatings (by coating cracks), and through the compacted cermet (by matrix pores).

Fuel loading and uranium nitride particle size. - Depending on the reactor application, it may be necessary to fabricate cermets with fuel loadings exceeding 50 volume percent of UN while still maintaining a continuous metal matrix and complete separation of the UN particles. Therefore, particles containing up to 70 volume percent of UN were consolidated into cermets by hot isostatic compaction. The microstructures of type Y W-UN cermets containing nominally 50, 60, and 70 volume percent of UN are shown in figure 9. Observe that the W matrix is continuous around the UN dispersoids in all these cermets. Additional examples of type X W-UN and Mo-UN cermets containing 70 volume
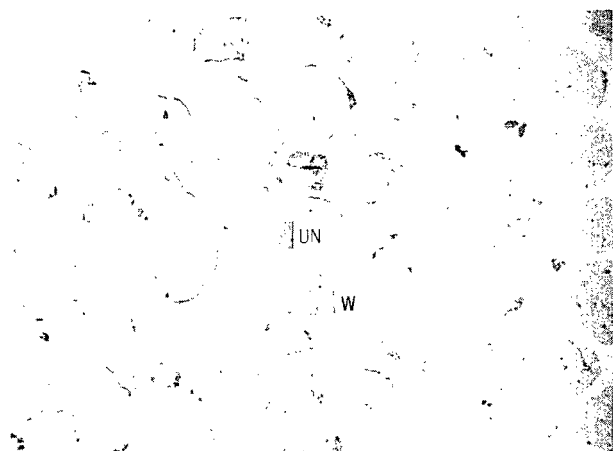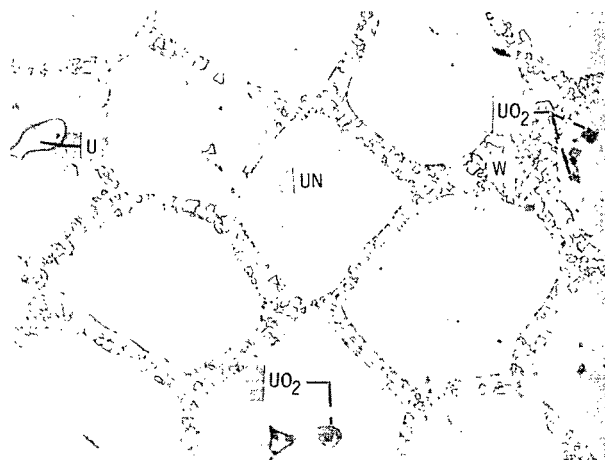
22
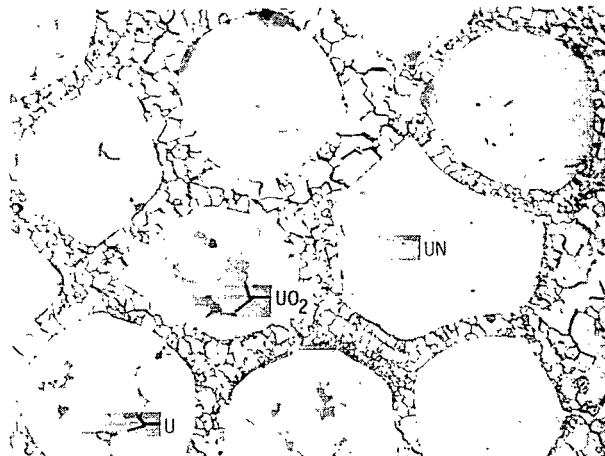
(a-1) X100.

(a-2) Etchant, Murikami's reagent. X250.
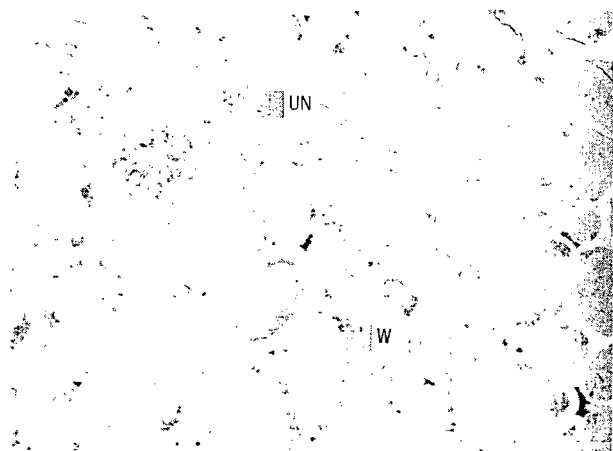
(a) Lot number, Y2W1; W - 50-volume-percent UN.
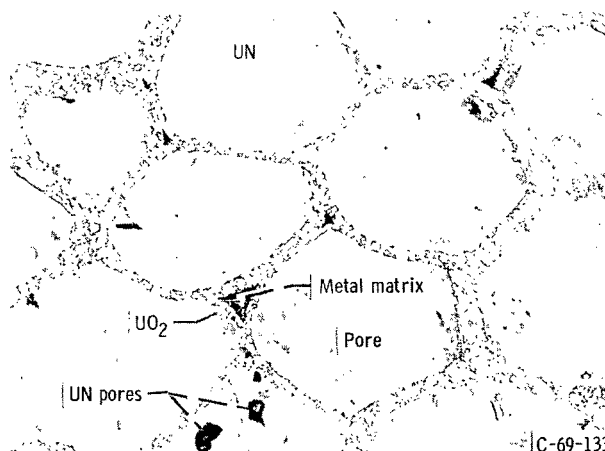
(b-1) X100.

(b-2) Etchant, Murikami's reagent. X250.

(b) Lot number, Y2W2; W - 60-volume-percent UN.
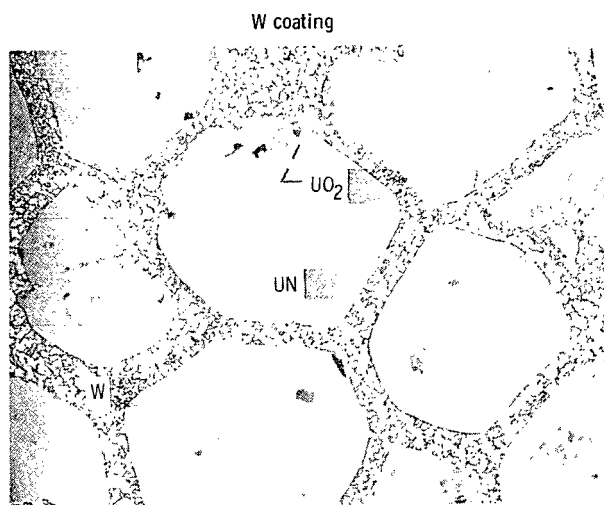
(c-1) X100.

(c-2) Etchant, Murikami's reagent. X250.

(c) Lot number, Y2W3; W - 70-volume-percent UN.

Figure 9. - Photomicrographs of cermets prepared from W-coated UN particles (75 to 150 μm diameter) containing 50 to 70 volume percent of UN.

W coating

Mo coating

(a) Lot number, X1W; UN particle diameter, 100 to 150 micrometers.
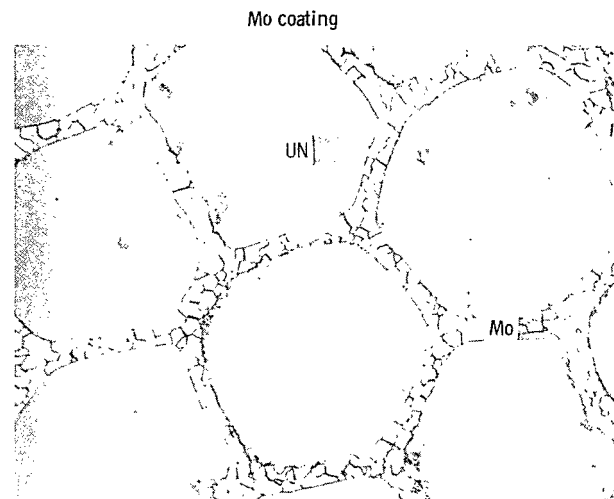
(b) Lot number, X1M; UN particle diameter, 100 to 150 micrometers.
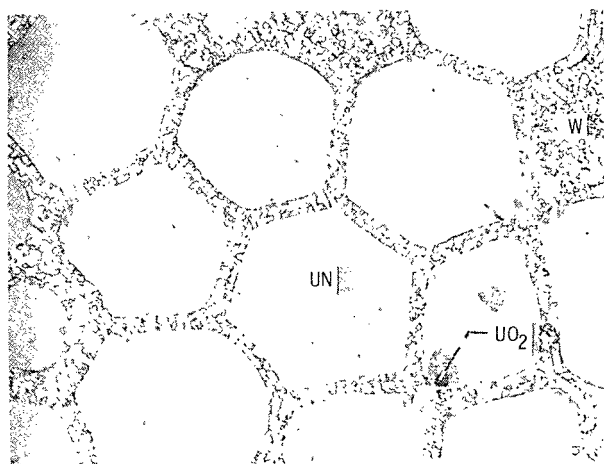
(c) Lot number, X2W; UN particle diameter, 75 to 100 micrometers.

(d) Lot number, X2M; UN particle diameter, 75 to 100 micrometers.

C-69-134

(e) Lot number, X3M; UN particle diameter, 100 to 150 micrometers.

Figure 10. - Photomicrographs of cermets containing 70 volume percent of UN. Etchant, Murikami's reagent. X250.

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percent of UN are shown in figure 10. Both these metals also formed a continuous matrix around all the UN dispersoids. As expected, Mo exhibited a larger grain size than W after hot pressing.

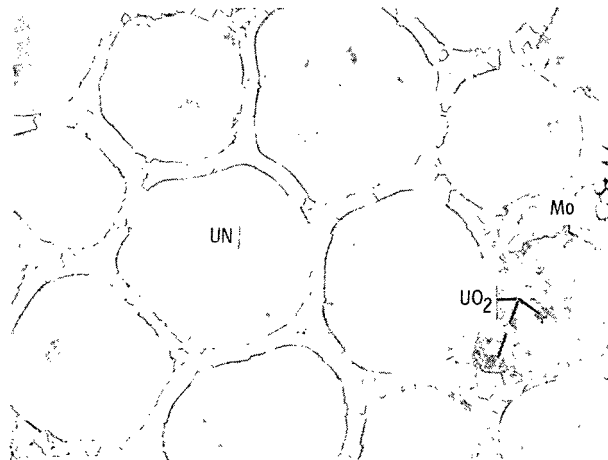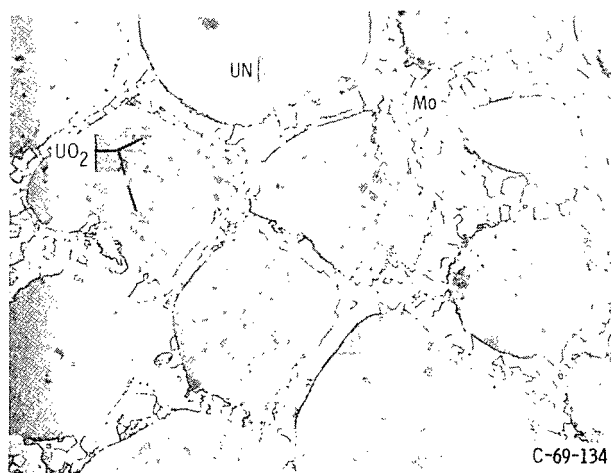Thermal gradients in UN particles in a cermet-type fuel element can be reduced by keeping the fuel dispersoids as small as possible consistent with maintaining a continuous metal matrix. The effect of reducing the UN dispersoid diameters from the range of 100 to 150 micrometers (figs. 10(a) and (b)) to the range of 75 to 100 micrometers (figs. 10(c) and (d)) on the interdispersoid spacing was demonstrated for 70-volume-percent UN cermets. A continuous metal matrix was achieved with both the larger and smaller sized dispersoids. However, if the dispersoid size became too much smaller, the interdispersoid spacing would decrease to a point where the metal matrix could become discontinuous. Thus, a minimum dispersoid size probably exists for a given fuel (UN) loading; this minimum size increases with increased fuel loadings.

Chemical compatibility of cermets. - Similar to the sintered cermets, no reaction between UN and W or Mo was observed in coated particles either before or after hot isostatic consolidation. Neither was any reaction observed between the cermet and the Mo containment can. Although the cermets were not chemically analyzed, metallographic examination revealed no significant change in either the UN stoichiometry (i.e., the free U present) or UO_2 content as a result of consolidation in high-temperature helium. This lack of change in the UN is attributed to the sealed Mo containment can which acted as a diffusion barrier to (1) prevent escape of N_2 from the cermet (and thus prevent UN decomposition) and (2) prevent oxidation of the UN by O_2 impurity in the pressurized helium.

COMPARISON OF CERMET FABRICATION METHODS

Two methods of fabricating W-UN and Mo-UN cermets are presented in this report, that is, cold-pressing and sintering of powder mixtures and hot isostatic compaction of coated, spherical UN particles. The purpose of this section of the report is to compare these methods of producing UN cermets.

Cold-pressing and sintering powder mixtures is by far the simpler and cheaper way of producing cermets. Neither the UN spheroidization process nor the particle-coating process are required for sintered cermets. These two processes are hard to control and give relatively low yields; therefore, at the present state of technology, they are relatively expensive processes. Another advantage of sintered cermets is that the dispersion of UN particles in the metal matrix can be much finer than is possible with coated particles for high fuel loadings. Also, it is fairly easy to control the amount of porosity in sintered cermets if this is required for vented-type fuel elements.

The sintering process also has disadvantages. Highly dense cermets can only be obtained by reducing the particle size of the UN (i. e., submicrometer). Unfortunately, the very fine UN powders generally contain much more UO_2 impurity because of the larger powder surface area and the reactivity of UN with oxygen.

Hot isostatic compaction, on the other hand, can produce highly dense cermets with larger, more UO_2-free particles. In addition, a better fuel distribution can be achieved with metal-coated spherical particles than with sintered cermets in which the metal tends to agglomerate. Coated particles also allow complete separation of the UN particles with much higher fuel (UN) loadings than is possible with powder mixtures.

In summary, the fuel element application (e.g., fuel burnup and density requirements) will largely dictate the method of fabricating UN cermets. At the present state of development, I believe that much more work is needed to perfect the process of fabricating coated-particle cermets than that of fabricating sintered powder cermets.

CONCLUSIONS

Two methods of fabricating uranium nitride (UN) cermets with tungsten (W) or molybdenum (Mo) matrixes were investigated. The first method consisted of sintering powder mixtures. The second method consisted of consolidating metal-coated UN particles by hot isostatic compaction.

The following conclusions are made regarding the fabrication and characterization of cold-pressed and sintered cermets containing 50 volume percent of UN:

1. The density of W and Mo cermets sintered in a nitrogen (N_2) atmosphere can be varied from 80 to 98 percent of theoretical by varying the metal or UN particle size and by varying the sintering temperature and time. Decreasing the particle sizes and increasing the sintering temperature or time tend to increase the density of cermets.

2. Neither W nor Mo chemically reacts with UN at temperatures up to 2200° C provided that N_2 is present in the sintering atmosphere. However, when N_2 is not present, cermets exhibit evidence of uranium liquid-phase formation and enhanced sintering. Molybdenum-UN cermets appear to be more susceptible than W-UN cermets to liquid-phase formation and liquid-phase sintering.

3. Most of the porosity in cermets sintered in N_2 is interconnected to the specimen surfaces provided that the cermet density does not exceed about 90 percent of theoretical.

4. Hot pneumatic impaction of presintered cermets results in nearly complete densification of the cermets without any liquid-phase sintering.

The following conclusions are made regarding the fabrication and characterization of cermets prepared by consolidating metal-coated UN particles:

1. Spherical UN particles (from 50 to 150 μm in diameter) exhibiting stoichiometric composition, low oxygen impurities ($<$1000 ppm), and high densities can be produced. However, problems in controlling the spheroidization process prevent production of consistently acceptable particles.

2. Spherical UN particles in several different size ranges can be coated with thin layers of W or Mo to achieve fuel loadings of 50 to 70 volume percent of UN. Although these coatings contain many discontinuities (e.g., cracks), these discontinuities disappear during subsequent particle consolidation.

3. Coated UN particles can be successfully consolidated by hot isostatic compaction into cermets with densities exceeding 97 percent of theoretical. These cermets exhibit continuous metal matrixes and complete separation of the UN particles at 50- to 70-volume-percent fuel (UN) loadings. The metal matrixes appear to be compatible with UN in all the cermets.

Lewis Research Center,
    National Aeronautics and Space Administration,
        Cleveland, Ohio, January 3, 1969,
            120-27-04-60-22.

APPENDIX – CHEMICAL ANALYSES OF TUNGSTEN URANIUM NITRIDE – AND MOLYBDENUM – URANIUM NITRIDE CERMETS

Generally accepted methods of chemical analyses in W-UN and Mo-UN cermets have not been developed to date. Therefore, a limited study was conducted to determine the precision of several methods of analyses and to compare the results of different methods.

Analyses were run on sintered cermets to determine their U, N_2, O_2, and C contents. After the cermets were crushed into powder, representative samples were obtained by the coning and quartering technique. Great care was taken to crush and store the samples in Ar to avoid O_2 contamination. In addition, the UN samples were sealed in tin capsules prior to chemical analysis to prevent O_2 contamination.

It is possible to calculate the UN stoichiometry (N/U) from the chemical analysis. For purposes of N/U calculations, all O_2 was assumed to be in the form of UO_2 in the UN. This assumption is reasonable because (1) UO_2 is more stable than the W oxides and (2) metallographic examinations revealed an increasing amount of UO_2 phase in the UN with increasing O_2 contents in the cermets. Calculations showed no significant change in the N/U regardless of whether or not C was assumed to be in the form of uranium carbide (UC) in the UN. Thus, C was ignored for purposes of the N/U calculations.

The results of the analyses are given in table VI for both W-UN and Mo-UN sintered cermets. Based on these analyses, the following results were obtained:

(1) The precision of the U analysis indicates that there is a 0.99 probability that an analysis is within 1 percent of the average value.

(2) The precision of the N_2 analysis indicates that there is a 0.99 probability that a Kjeldahl analysis is within 3 percent of the average value and that a vacuum fusion analysis is within 5 percent of the average value. The two methods of analysis for N_2 yielded average values within 1 percent of each other; this close agreement lends support to the accuracy of analyses.

(3) The precision of the O_2 analyses indicates that there is a 0.99 probability that both the vacuum fusion and inert gas fusion analyses are within 5.5 percent of the average value for W-UN cermets. For Mo-UN cermets, vacuum fusion is within 9 percent and inert gas fusion is within 7.5 percent of the average value. The two methods of analysis yielded average values within 1 percent of each other for the W-UN cermet. But the average values were 20 percent different for the Mo-UN cermet. Either Mo interfers with one or both methods of O_2 analysis or the vacuum fusion samples were inadvertently contaminated with O_2.

(4) The precision of the C analysis indicates that there is a 0.99 probability that an analysis is within 7 to 11 percent of the average value.

TABLE VI. - CHEMICAL ANALYSIS OF TUNGSTEN - URANIUM NITRIDE

AND MOLYBDENUM - URANIUM NITRIDE CERMETS

Cermet material[a]	Element analyzed, concentration unit	Analysis method	Number of samples	Results of analyses			
				Range	Average	Standard deviation	99 Percent confidence limits[b]
W-UN	U, wt. %	Ion exchange	6	37.99 to 38.59	38.31	0.23	±0.33
	N, wt. %	Kjeldahl	6	2.16 to 2.27	2.20	0.045	±0.06
		Vacuum fusion	6	2.11 to 2.28	2.18	0.071	±0.10
	O, ppm	Vacuum fusion	6	3180 to 3470	3358	135	±185
		Inert gas fusion	7	3240 to 3610	3400	155	±184
	C, ppm	Combustion	6	50 to 59	54	3.0	±4
Mo-UN	U, wt. %	Ion exchange	6	54.19 to 54.79	54.49	0.22	±0.31
	N, wt. %	Kjeldahl	6	2.88 to 3.05	2.96	0.064	±0.9
		Vacuum fusion	5	2.89 to 3.10	2.99	0.092	±0.15
	O, ppm	Vacuum fusion	5	3310 to 3580	3424	183	±307
		Inert gas fusion	7	2500 to 3040	2724	202	±240
	C, ppm	Combustion	6	46 to 58	52	4.5	±6

[a]Contained nominally 50 vol. % of UN; sintered for 3 hr at 2200° C in Ar (33 vol. %) - N_2 (67 vol. %) prior to analysis.

[b]Based on the small sample "t-distribution" calculation.

Based on the preceding results, one can conclude that the N/U for W-UN and Mo-UN cermets is within 3 to 4 percent of the value calculated. These results also indicate the precision and accuracy of the analytical methods used. Other than the O_2 analysis on the Mo-UN cermet, the precision and accuracy of the analyses methods appear to be quite good. However, these results do not imply that the same precision and accuracy could be achieved with different fuel loadings, lower O_2 contents, etc. Much more work is required to establish the validity of these results and the precision and accuracy of the analytical methods.

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